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Certificate

REPUBLIC OF SOUTH AFRICA

PATENT OFFICE
DEPARTMENT OF TRADE AND
INDUSTRY

Hiermee word gesertifiseer dat
This is to certify that

- 1) South African Patent Application No. **2003/2620** accompanied by a Provisional Specification was filed at the South African Patent Office on **3 April 2003** in the name of **Merisol (RSA) (Pty) Limited** in respect of an invention entitled: "**Purification of phenols**".
- 2) The photocopy attached hereto is a true copy of the provisional specification filed with South African Patent Application No. **2003/2620**.

Geteken te

PRETORIA

Signed at

in die Republiek van Suid-Afrika, hierdie

in the Republic of South Africa, this

dag van

5th

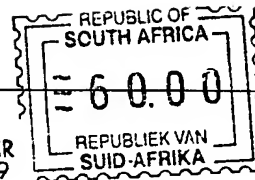
April 2004

day of

Registrar of Patents

REPUBLIC OF SOUTH AFRICA				PATENTS ACT, 1978			
REGISTER OF PATENTS							
OFFICIAL APPLICATION NO.			LODGING DATE : PROVISIONAL			ACCEPTANCE DATE	
21	042003/2620		22	3 April 2003		43	
INTERNATIONAL CLASSIFICATION			LODGING DATE : COMPLETE			GRANTED DATE	
51			23				
FULL NAME(S) OF APPLICANT(S) / PATENTEE(S)							
71	MERISOL (RSA) (PTY) LIMITED						
APPLICANTS SUBSTITUTED :						DATE REGISTERED	
71							
ASSIGNEE(S)						DATE REGISTERED	
71							
FULL NAME(S) OF INVENTOR(S)							
72	LUND, Grant James						
	COOGAN, Edward John						
	JANSEN, Wilhelmina						
PRIORITY CLAIMED		COUNTRY		NUMBER		DATE	
N.B. Use international abbreviation for country. (See Schedule 4)		33		31		32	
TITLE OF INVENTION							
54	PURIFICATION OF PHENOLS						
ADDRESS OF APPLICANT(S) / PATENTEE(S)							
2 Sturdee Avenue Rosebank Johannesburg South Africa							
ADDRESS FOR SERVICE						REF	
74	D M Kisch Inc, 54 Wierda Road West, Wierda Valley, SANDTON					P26465ZA00	
PATENT OF ADDITION NO.			DATE OF ANY CHANGE				
61							
FRESH APPLICATION BASED ON			DATE OF ANY CHANGE				

REPUBLIC OF SOUTH AFRICA
PATENTS ACT, 1978



Form P.1

APPLICATION FOR A PATENT AND ACKNOWLEDGEMENT OF RECEIPT
(Section 30 (1) - Regulation 22)
The grant of a patent is hereby requested by the undermentioned applicant on the basis of the present application filed in duplicate.

OFFICIAL APPLICATION NO	
21	01 2003/2620

DMK REFERENCE
P26465ZA00

FULL NAME(S) OF APPLICANT(S)

71	MERISOL (RSA) (PTY) LIMITED
----	-----------------------------

ADDRESS(ES) OF APPLICANT(S)

	2 Sturdee Avenue Rosebank Johannesburg South Africa
--	--

TITLE OF INVENTION

54	PURIFICATION OF PHENOLS
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	THE APPLICANT CLAIMS PRIORITY AS SET OUT ON THE ACCOMPANING FORM P2 The earliest priority claimed is
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	THIS APPLICATION IS FOR A PATENT OF ADDITION TO PATENT APPLICATION NO.	21	01	
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	THIS APPLICATION IS FRESH APPLICATION IN TERMS OF SECTION 37 AND BASED ON APPLICATION NO.	21	01	
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THIS APPLICATION IS ACCOMPANIED BY :	
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<input checked="" type="checkbox"/>	1a	A single copy of a provisional specification of 17 pages.		
	1b	Two copies of a complete specification of pages.		
	2a	Informal drawings of sheets.		
<input checked="" type="checkbox"/>	2b	Formal drawings of 1 sheets.		
	3	Publication particulars and abstract (form P8 in duplicate).		
	4	A copy of figure of the drawings for the abstract.		
	5	Assignment of invention (from the inventors) or other evidence of title.		
	6	Certified priority document(s).		
	7	Translation of priority document(s).		
	8	Assignment of priority rights.		
	9	A copy of form P2 and a specification of S.A. Patent Application.	21	01
	10	A declaration and power of attorney on form P3.		
	11	Request for ante-dating on form P4.		
	12	Request for classification on form P9.		
	13a	Request for delay of acceptance on form P4.		
	13b			

DATED 3 April 2003

ADDRESS FOR SERVICE	
74	D M Kisch Inc Inanda Greens Business Park 54 Wierda Road West Wierda Valley SANDTON

Patent Attorney for Applicant(s)

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REGISTRATEUR VAN PATENTE, MODELLE, HANDELSMERKE EN OUTEURSREG	

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REPUBLIC OF SOUTH AFRICA

PATENTS ACT, 1978

PROVISIONAL SPECIFICATION
(Section 30 (1) - Regulation 27)

OFFICIAL APPLICATION NO.		LODGING DATE		DMK REFERENCE
21	01	22	3 April 2003	P26465ZA00
2003/2620				
FULL NAME(S) OF APPLICANT(S)				
71	MERISOL (RSA) (PTY) LIMITED			
FULL NAME(S) OF INVENTOR(S)				
72	LUND, Grant James COOGAN, Edward John JANSEN, Wilhelmina			
TITLE OF INVENTION				
54	PURIFICATION OF PHENOLS			

TECHNICAL FIELD

This invention relates to the purification of phenols in a feedstock containing
5 phenols and neutral oils and/or tar bases.

BACKGROUND ART

Feedstocks containing mixtures of phenols (such as phenol, cresols,
10 ethylphenols, xylenols and C3 phenols) and neutral oils and/or tar bases are
obtained from various sources such as petroleum and coal processing facilities.

The separation of phenols from tar bases and especially, neutral oils have
always posed a challenge since certain neutral oils have boiling points close to
15 that of the phenols and due to the fact that azeotropes are formed between the
neutral oils and phenols. Normal distillation processes are accordingly not
suitable to separate phenols from neutral oils and/or tar bases to the required
purity.

20 Liquid-liquid extraction is a known method of separating phenolics from
neutral oils and/or tar bases. The prior art discloses that both single solvent
and dual solvent processes have been employed to achieve such a
separation. Dual solvent processes are preferred. In these processes a
solvent is used to extract the phenolics and a counter-solvent is used to
25 extract the neutral oils. The solvent and counter-solvent are selected to be

immiscible and after extraction the solvent and counter-solvent layers are separated and the phenolics are recovered from the solvent layer while the neutral oils are recovered from the counter-solvent layer.

The solvent usually is a polar solvent and the counter-solvent usually is a non-polar solvent. The counter-solvent often comprises paraffins such as hexane, heptane, petroleum ether, octane and diesel. The polar solvent often comprises an aqueous solution of methanol, ammonia, acetamide, acetic acid, ethanol and glycols. In this regard see for example US Patent 2,666,796 where aqueous methanol and hexane were used as solvent and counter-solvent respectively. In the Journal of Applied Chemistry, 21 June 1952, page 314, and the Journal of Applied Chemistry, 3, March, 1953, page 98, aqueous triethylene glycol was used as solvent. In Fuel processing Technology, 28 (1991) page 287 aqueous solutions of ethylene glycol, diethylene glycol and triethylene glycol were used as solvents. In South African patents 98/11312 and 2001/9287 aqueous solutions of glycols were used as solvents and they were used in combination with counter-solvents.

In cases where the solvent comprises a mixture of water and a solvent compound (e.g. glycols) a solvent layer is obtained which contains the water, the solvent compound (e.g. glycols), phenolics, some neutral oils and/or tar bases and some counter-solvent. The first step is usually to subject the mixture to a water recovery distillation process to strip the

water from this mixture. The bottoms product consists mainly of the solvent compound (e.g. glycols) and phenolics, which bottoms product is then subjected to distillation to separate the phenolics from the glycols.

5 In South African patent 2001/9287 the solvent comprised a mixture of glycol and water and the water recovery distillation process was carried out under vacuum to prevent thermal decomposition of the glycol. The overheads product of the water recovery distillation process (consisting of water, some counter-solvent, some phenols and some neutral oils and/or tar
10 bases) was condensed and was then mixed with counter-solvent to extract the condensate with the counter-solvent. A decanter was then used to achieve separation of the aqueous and organic layers which formed. It was suggested that the counter-solvent added resulted in extraction of organic material into the counter-solvent layer and it also resulted in better phase
15 separation in the decanter.

South African patent 98/11312 discloses a similar water recovery step to that disclosed in South African patent 2001/9287 except that no counter-solvent was added to the overheads product of the water recovery
20 distillation process.

In both South African patents 98/11312 and 2001/9287 the separated organic layer from the overheads of the water recovery distillation process

was routed to the recovery of the counter-solvent from the counter-solvent layer formed by the liquid-liquid extraction process. In both South African patents 98/11312 and 2001/9287 the counter-solvent recovery was achieved by subjecting the counter-solvent layer to an counter-solvent
5 recovery distillation process to yield recovered counter-solvent (usually as an overheads product) and neutral oils and/or tar bases (usually as a bottoms product). The recovered counter-solvent was then recycled to the liquid-liquid extraction step and the neutral oils and/or tar bases were then routed to a neutral oils and/or tar bases processing facility.

10

It has now been established that in certain cases the overheads product of the water recovery distillation process contains significant concentration of phenols which form part of the organic layer which is separated from the aqueous layer of the overheads product of the water recovery distillation
15 process (particularly when operating the extract stripper or water recovery distillation under pressure). In the prior art processes of South African patents 98/11312 and 2001/9287 these phenols were lost since they were routed to the counter-solvent recovery process where the phenols ended up with the neutral oils and/or tar bases which were routed to the neutral oils
20 and/or tar bases processing facility.

It is an object of the present invention to at least reduce the above disadvantage.

DISCLOSURE OF THE INVENTION

According to the present invention a process for purifying phenols from a feedstock containing phenols, neutral oils and/or tar bases comprises the
5 steps of

- 10 i) subjecting the feedstock to liquid-liquid extraction using a solvent in the form of mixture of water and a solvent compound to transfer the phenols into a solvent layer, and using an organic counter-solvent to transfer the neutral oils and/or tar bases into a counter-solvent layer;
- ii) separating the solvent and counter-solvent layers;
- 15 iii) subjecting the separated solvent layer of step ii) to distillation to yield a mixture comprising recovered water, some counter-solvent and some phenols as an overheads product, and a mixture comprising the solvent compound and a major portion of the phenols as a bottoms product;
- 20 iv) settling the overheads product of step iii) to form an aqueous phase and an organic phase and separating the phases from each other;
- v) recycling the organic phase of step iv) to the liquid-liquid extraction of step i); and
- vi) recovering the phenols from the bottoms product of step iii) to

yield a purified phenols product.

Preferably the distillation of step iii) is operated at or above atmospheric pressure. Preferably the distillation of step iii) is operated at a pressure above
5 0 kPag, preferably from 0 to 100 Kpag, preferably at about 20kPag. The distillation of step iii) is preferably carried out at a temperature at which the solvent compound and preferably also the counter-solvent will not decompose. In the case where triethylene glycol is used as a solvent the temperature may be from 150°C to 207°C, preferably about 195°C. The distillation of step iii) is
10 preferably also performed under such conditions in order that the water concentration is larger than 50wt% throughout the distillation process preferably greater than 70wt%. A distillation column may be used to perform the separation and preferably the water concentration is larger then 50wt%, preferably larger than 70wt% throughout the distillation column, excluding the
15 re-boiler.

Operating the distillation of step iii) under such conditions will result in some water ending up in the bottoms product of the distillation. This water can be separated out, if required, during later processing steps.

20

In a preferred embodiment of the invention the separated solvent layer of step (ii) is mixed with counter-solvent for extracting neutral oils and/or tar bases therein during or before the distillation of step iii). Preferably the counter-solvent is added to the feed of the distillation of step iii), that is addition prior to

the said distillation. Preferably the counter-solvent is added at a mass ratio of 0.0 to 0.5 to the separated solvent layer fed to the distillation of step iii), preferably at about 0.04.

- 5 The solvent compound preferably also comprises an organic compound.

The overheads product of step iii) may be condensed before it is settled and separated according to step iv). The separation of step iv) may be by decanting.

10

The separated organic phase of step iv) is recycled to the liquid-liquid extraction step. This organic phase will usually contain counter-solvent; some neutral oils and/or tar bases and nearly all of the phenols which report to the overheads during the distillation step. By recycling this phase to the liquid-liquid extraction step the phenols and counter-solvent contained therein are not lost. In the prior art the phenols were lost.

15

The separated aqueous phase of step iv) may be purged or recycled.

20

By operating the distillation of step iii) under atmospheric or higher pressures the disadvantages of vacuum distillation is avoided or at least reduced. Such a vacuum distillation would cause major losses of counter-solvent to the vacuum system used to create the vacuum. It also improves control of the distillation, but it particularly causes improved separation of the organic and aqueous

phases in the overheads product mixture (ie. no counter-solvent is required to be added after condensation of the overheads to step iii).

5 The addition of counter-solvent to the distillation of step iii) also improves recovery of phenols to the said organic phase. It further improves stripping of neutral oils and/or tar bases during the distillation of step iii). Improved capital and operating costs are also achieved.

10 Recovery of phenols of step vi) may be achieved by distillation, preferably distillation under vacuum. Water usually forms part of the bottoms product mixture of step iii) and at least some of this water ends up in the vacuum system, used to create a vacuum when the phenols are recovered under vacuum according to step vi). The vacuum system is preferably operated in order to ensure that at least 50wt%, but preferably most of the water is lost to
15 the vacuum system. This assists in drying the phenols.

However, phenols will also be lost to the vacuum system, but this loss can be minimized by recycling water with phenols therein recovered from the vacuum system, for example to the distillation of step iii). The water recovered from the
20 vacuum system may be recycled to the feed stream to the distillation of step iii).

The separated counter-solvent layer of step ii) may be subjected to separation of the counter-solvent and the neutral oils and/or tar bases. The counter-solvent and neutral oils and/or tar bases may be separated by distillation,

preferably by distillation under pressure. Preferably the counter-solvent is selected to be light boiling relative to the neutral oils and/or tar bases and thus the counter-solvent goes to the overheads product during separation by distillation. The recovered counter-solvent may be recycled to the liquid-liquid
5 extraction step.

The counter-solvent layer of step ii) will usually contain at least some solvent compound (usually due to the solubility of the solvent compound in the counter-solvent) and it will usually also contain some phenols. Although the amount of
10 solvent in the separated counter-solvent layer of step ii) can be adjusted by adjusting operation conditions of the liquid-liquid extraction, such adjustments impact on the effectiveness of the liquid-liquid extraction. The solvent in the separated counter-solvent layer will end up in the bottoms product with the neutral oils and/or tar bases if distillation is used to separate the counter-solvent
15 from the counter-solvent layer. In the prior art such solvent was lost. However, this bottoms product may now be treated to separate the solvent from the neutral oils and/or tar bases. Preferably the said bottoms product from the counter-solvent distillation separation is mixed with water and which mixture is then subjected to phase separation to obtain an organic phase containing the
20 neutral oils and/or nitrogen bases and an aqueous phase containing the water and some solvent compound and usually also some phenols. The aqueous phase may then be recycled to the liquid-liquid extraction step, preferably by adding it to the solvent to be added to the liquid-liquid extraction step. The phase separation may be achieved by a liquid-liquid extraction unit, but

preferably the mixture of phases is allowed to settle causing phase separation, which phases are then separated, for example, de-canted.

The liquid-liquid extraction may comprise counter-current liquid-liquid extraction
5 and a fractional liquid-liquid extraction may be used.

The solvent mixture may comprise a mixture of water and polar solvent, preferably a glycol. Preferably the glycol comprises triethylene glycol (TEG). The mass ratio of water to glycol may be from 0.1 to 0.3 preferably it is about
10 0.2. The mass ratio of solvent mixture to feedstock may be from 0.3 to 3, preferably about 0.7

The counter-solvent may comprise a non-polar compound, preferably it comprises a paraffinic compound on a mixture of paraffinic compounds (e.g.
15 paraffins and isoparaffins). In one embodiment of the invention the counter-solvent may comprise hexane. The mass ratio of counter-solvent to feedstock may be from 0.5 to 3.0, preferably about 1.8.

Preferably the solvent compound is low boiling relative to the neutral oils and/or
20 tar bases, and preferably the counter-solvent is high boiling relative to the neutral oils and/or tar bases.

The phase separation of step ii) may be achieved by any convenient manner e.g. decanting or during fractional extraction.

The invention also relates to phenols purified according to the process.

The invention will now be further described by means of the attached Figure
5 which is a diagrammatic layout of a process according to the invention.

Referring now to the Figure a method of purifying phenols in a feedstock stream
10 containing phenols, neutral oils and tar bases comprises as a first step
subjecting the feedstock stream 10 to liquid-liquid extraction (LLE) in a liquid-
10 liquid extraction unit 11. The feedstock stream 10 was a concentrated phenols
mixed feed stream comprising 90 wt% phenols, the remainder being neutral oils
and nitrogen bases in the naphtha/medium creosote range. This feedstock
stream 10 was mixed with a feedstream of naphtha 12 having a phenolic
content of ca. 31 wt% at a mass ratio of 0.3 to the feed stream 11. The
15 concentrated phenolic stream is cleaned up in the process while the dilute
phenolic stream (feed stream 12, naphtha) allows for the production of
additional phenolics.

Counter solvent in the form of hexane was fed via stream 13 to the bottom of
20 the LLE unit 11 at a mass ratio of 1.8 to the combined feed streams 10 and 12 .

Other non-polar counter-solvents in the form of paraffinic compounds or
mixtures thereof may be used in other applications. Solvent in the form of a
mixture of water and a solvent compound in the form of triethylene glycol (TEG)
in a mass ratio of 0.2 water/0.8 TEG. was fed via stream 14 to the LLE unit 11

at the top thereof. The solvent feed 14 was fed at a mass ratio of 0.7 to the combined feeds 10 and 12. Other polar solvents, especially aqueous polar solvents may be used in other cases.

- 5 The LLE unit 11 can be any conventional LLE unit (e.g. packed, Karr, rotating disc contactor), but in this case it was a Scheibel type counter-current unit. The impeller diameter of the unit 11 was different throughout the length of the column to maximize mass transfer and throughput before flooding commences.

The LLE unit 11 may be operated at various temperature (e.g. 25 to 70°C) but
10 in this particular example it was operated at a temperature of 45°C.

The raffinate stream 15 of the LLE unit 11 contained counter-solvent, and smaller amounts of phenols and TEG (due to its solubility in the counter-solvent). The raffinate stream 15 was fed to a distillation column (or raffinate stripper) 16 to recover the counter-solvent as an overheads product stream 17
15 which was condensed in a condenser 18 and transferred to a reflux drum 19.

The recovered counter-solvent was recycled via stream 20 to the LLE unit 11. Counter-solvent make up may be added via stream 21 to the reflux drum 19 as this gives a quick response to surges. Alternatively [not shown], counter-solvent make-up may be added to the counter-solvent feed stream 13. The distillation
20 column 16 may be run at 0-200kPag pressure and a temperature profile from 55 to 230° C. In this example the pressure was 70kPag and the reboiler temperature was 215°C.

The bottoms product stream 22 of the distillation column 16 was rich in neutral

oils and tar bases, but also included some TEG and some phenols. This bottoms product stream 22 was mixed with water at a ratio of 0.4, which water was from stream 23 (which will be described later). The resultant mixture stream 24 was fed to a decanter 25 where it phase separated into an organic layer stream 26 of neutral oils and tar bases which was further worked up [not shown]; and an aqueous layer stream 27 containing water and recovered TEG and some recovered phenols. The stream 27 was recycled to the LLE unit 11, particularly to the solvent feed stream 14. The one main advantage of addition of water via stream 23 and the separation in 25 is that TEG is recovered, which is then recycled. The use of water via stream 23 also minimizes the water balance (i.e. purging of water). In the decanter 25 the temperature was 55°C and the pressure was 15kPag, but other temperatures e.g. 25 to 75°C and other pressures may also be used.

The bottoms stream 28 from the LLE unit 11 mainly comprised solvent compound (TEG), water, phenols, some counter-solvent, and some neutral oils and tar bases. The stream 28 was fed to a distillation column or extract stripper 29 and it was mixed with a slipstream 30 of counter-solvent and a stream 34 waste water from a vacuum system, in particular steam ejectors (not shown, but described later) at a mass ratio of 0.04 counter solvent and 0.04 water from ejectors to total feed to extract stripper. The extract stripper 29 was operated under pressure, namely a pressure of 30 kpag. Some water was removed with the solvent compound (TEG) and phenols via the bottoms stream 32. The water dramatically decreased the reboiler [not shown] temperature which

prevented TEG degradation. Although most of the water was removed via the overheads stream 33 (which contained water, some counter-solvent [which was light boiling], some neutral oils and tar bases, and some azeotroped phenols) the unit 29 no longer operated as a pure water recovery unit but as an extract
5 stripper as it stripped the remaining neutral oils or tar bases from the phenols and the solvent compound.

The overheads stream 33 was condensed by means of the condenser 34 and the condensate was then phase separated in the reflux drum/decanter 35 into
10 an aqueous layer stream 36 and an organic layer stream 37. The aqueous layer stream 36 was essentially free of phenols, neutral oils and tar bases and was partly fed via stream 38 as reflux to the stripper 29. Some of the remaining water, that is in stream 39, was worked up and some of this was purged to close the water mass balance, while some water, that is in stream 23, was mixed with
15 the stream 22 as described above.

The organic layer stream 37 comprised counter-solvent, phenolics, neutral oils, tar bases and was recycled to the LLE unit 11, particularly to the naphtha feed stream 12. (Ratio ca. 0.2:1 (mass)). Alternatively it may be added to the
20 counter-solvent feed stream 13 or the solvent feed stream 14, but the latter is not suggested. Since the stripper 29 was operated under pressure measurable amounts of phenols were lost to the overheads stream 33, but it was not lost to the process as it was recycled to the LLE unit 11 via stream 37.

Addition of the counter-solvent via stream 30 to the bottoms stream 28 prior to the stripper 29 improved phase separation in the decanter 35. Running the stripper 29 under pressure allowed one to add counter-solvent to the stripper 29 without loss of the counter-solvent to a vacuum system (as in the prior art).

5 More phenols were removed overhead via stream 33 but it was recovered via the recycling of the organic layer stream 37. Pressure operation of the stripper 29 gave more control to the stripper 29 and allowed for improved operation of the reflux drum/decanter 35.

10 The bottoms stream 32 contained solvent compound (TEG), phenols and some water and was forwarded to a distillation column or product stripper 40 which operated under vacuum to strip the phenols as an overheads stream 41. The vacuum system [not shown] was run to ensure that at least 50%wt, but preferably more water was lost to the vacuum system via stream 42. Dirty solvent product was recovered as the bottoms stream 43. The unit 40 was
15 operated under vacuum because of the solvent compound (TEG) degradation temperature and the stream saturation temperature. Typically the operating pressure is 3,5kPa abs and typically the bottoms temperature is <195°C, usually about 195°C.

20

The dirty solvent stream 43 was recycled back to LLE unit 11 especially to the solvent the solvent feedstream 14. A small slip stream 44 was cleaned by means of flash evaporation in the unit 45. The clean solvent stream 46 was mixed with the dirty solvent stream 43. The bottoms product stream 47 was

removed to be processed further. The flash evaporation 45 was typically run at 2kPa abs and at a temperature of 167°C.

5 The vent 48 of the flash was recovered in the vacuum system [not shown] which was recovered as a waste water stream 31, which was returned to the stripper 29.

10 The addition of the waste water stream 31 from the vacuum system of unit 40 means that phenols lost to the vacuum system may be recovered in the stripper 29. The waste water stream 31 may be added to the reflux drum/decanter 35 but it is not recommended as it may contain solvent which will be purged via the stream 39.

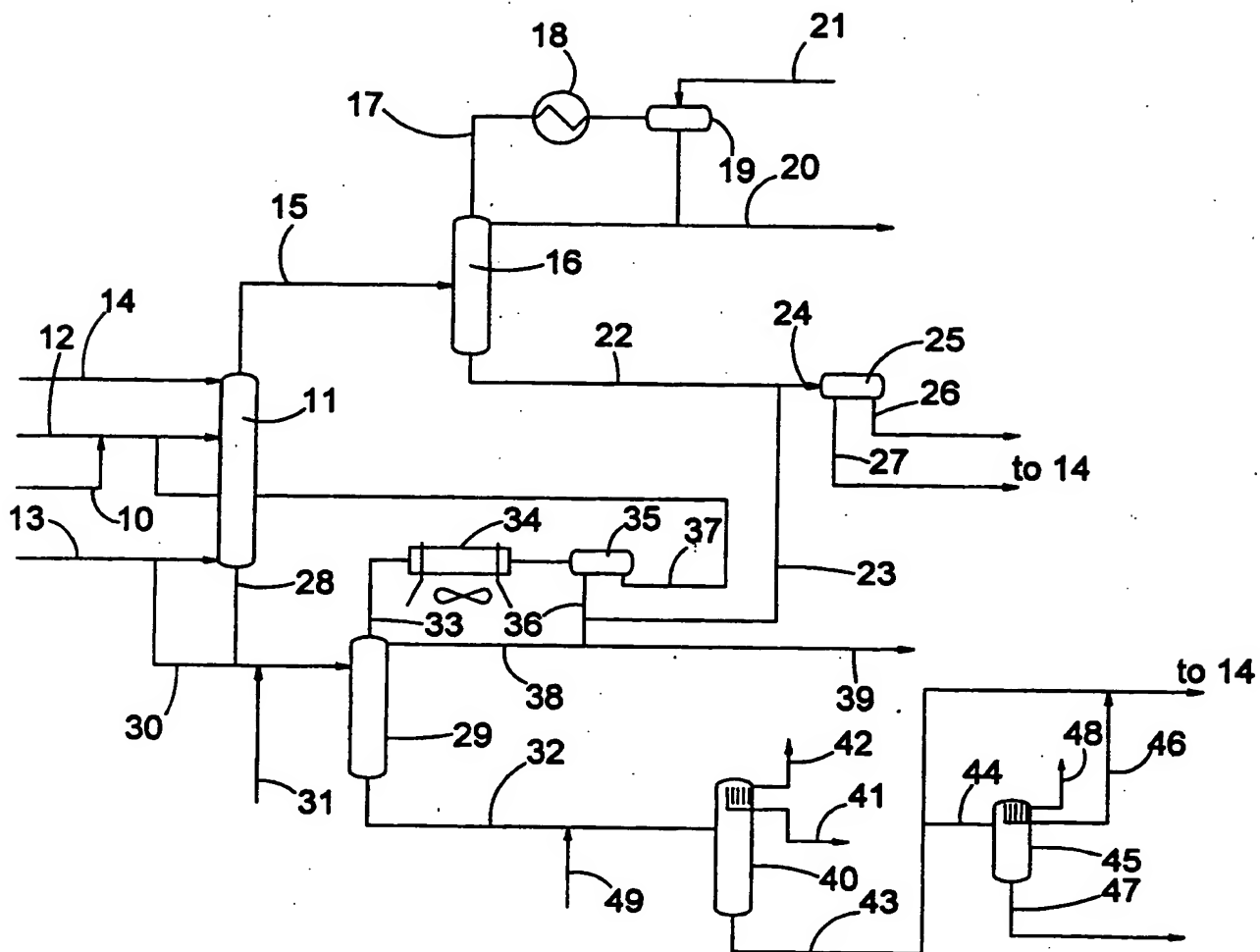
15 Make up TEG can be added as stream 49.

The separated phenols of stream 41 had a composition of 95%+ phenols, and ca. 1.6 wt% water. If required the water may be removed in a subsequent step by means of for example a distillation unit.

20 It will be appreciated that many variations in detail are possible without thereby departing from the scope and spirit of the invention.

Dated this 3 day of April 2003

16 Patent Attorney / Agent for the Applicant



FIGURE